

3-Methoxy-2-[5-(naphthalen-2-yl)-4,5-di-hydro-1H-pyrazol-3-yl]phenol

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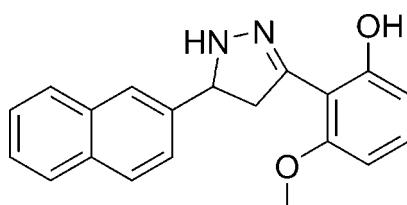
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Key indicators: single-crystal X-ray study; $T = 147\text{ K}$; mean $\sigma(\text{C}-\text{C}) = 0.002\text{ \AA}$; R factor = 0.037; wR factor = 0.097; data-to-parameter ratio = 12.2.

The asymmetric unit of the title compound, $C_{20}H_{18}N_2O_2$, contains two independent molecules in which the dihedral angles between the naphthalene ring system [r.m.s. deviations = 0.012 (1) and 0.015 (1) \AA] and the benzene ring are 71.65 (6) and 74.51 (6) $^\circ$. In the crystal, pairs of $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds form two independent inversion dimers with graph-set notation $R_2^2(14)$. In addition, each molecule contains an intramolecular $\text{O}-\text{H}\cdots\text{N}$ hydrogen bond with an $S(6)$ motif.

Related literature

For the synthesis and biological properties of pyrazoline derivatives, see: Hwang *et al.* (2013); Sharifzadeh *et al.* (2013); Congiu *et al.* (2010); Khode *et al.* (2009); Karthikeyan *et al.* (2007). For related structures, see: Fun *et al.* (2012); Jasinski *et al.* (2010). For hydrogen-bond graph-set notation, see: Bernstein *et al.* (1995).



Experimental

Crystal data

$C_{20}H_{18}N_2O_2$

$M_r = 318.36$

Monoclinic, $P2/c$

$a = 21.0215 (15)\text{ \AA}$

$b = 5.6564 (5)\text{ \AA}$

$c = 28.785 (2)\text{ \AA}$

$\beta = 110.543 (3)^\circ$

$V = 3205.1 (4)\text{ \AA}^3$

$Z = 8$
 $\text{Cu } K\alpha$ radiation
 $\mu = 0.69\text{ mm}^{-1}$

$T = 147\text{ K}$
 $0.17 \times 0.10 \times 0.07\text{ mm}$

Data collection

Bruker Kappa APEX DUO CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2012)
 $T_{\min} = 0.695$, $T_{\max} = 0.753$

43387 measured reflections
5506 independent reflections
4776 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.045$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.037$
 $wR(F^2) = 0.097$
 $S = 1.05$
5506 reflections
451 parameters

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\max} = 0.17\text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.20\text{ e \AA}^{-3}$

Table 1
Hydrogen-bond geometry (\AA , $^\circ$).

| $D-\text{H}\cdots A$ | $D-\text{H}$ | $\text{H}\cdots A$ | $D\cdots A$ | $D-\text{H}\cdots A$ |
|------------------------------------|--------------|--------------------|-------------|----------------------|
| O1A—H1OA \cdots N1A | 0.97 (2) | 1.71 (2) | 2.5754 (15) | 145 (2) |
| O1B—H1OB \cdots N1B | 0.91 (2) | 1.71 (2) | 2.5377 (15) | 149 (2) |
| N2A—H2NA \cdots O1A ⁱ | 0.914 (18) | 2.234 (18) | 3.0470 (16) | 147.7 (15) |
| N2B—H2NB \cdots O1B ⁱ | 0.911 (19) | 2.131 (19) | 2.9787 (16) | 154.4 (15) |

Symmetry codes: (i) $-x + 1, -y + 1, -z + 1$; (ii) $-x, -y - 2, -z + 1$.

Data collection: *APEX2* (Bruker, 2012); cell refinement: *SAINT* (Bruker, 2012); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

Supporting information for this paper is available from the IUCr electronic archives (Reference: IS5346).

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supplementary materials

Acta Cryst. (2014). E70, o464 [doi:10.1107/S1600536814005972]

3-Methoxy-2-[5-(naphthalen-2-yl)-4,5-dihydro-1*H*-pyrazol-3-yl]phenol

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1. Comment

Pyrazolines have been reported to show a broad spectrum of biological activities including antibacterial (Sharifzadeh *et al.*, 2013), anticonvulsant (Karthikeyan *et al.*, 2007), analgesic (Khode *et al.*, 2009) and antitumor properties (Congiu *et al.*, 2010). In continuation of our research interest to develop novel pyrazoline derivatives which show broad range of biological activities (Hwang *et al.*, 2013), the title compound (I) was synthesized and its crystal structure was determined.

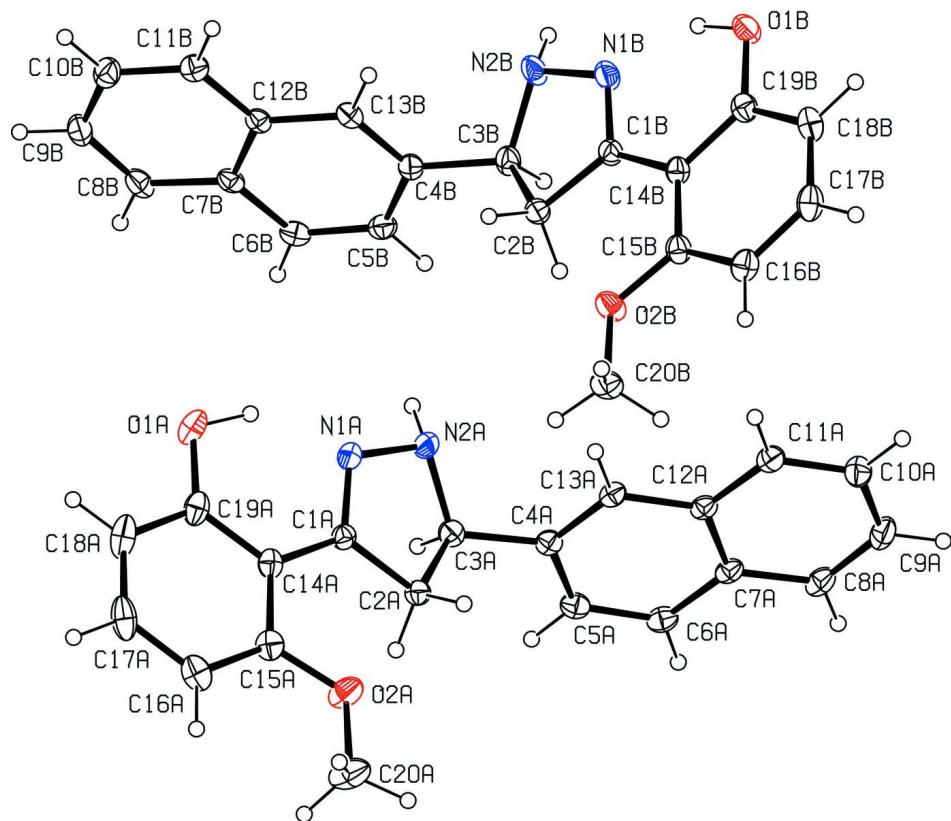
The asymmetric unit of (I) contains two independent molecules (A and B in Fig. 1). The dihedral angles between the naphthalene ring system [r.m.s. deviations 0.012 (1) Å for A and 0.015 (1) Å for B] and the benzene ring are 71.65 (6) and 74.51 (6)° for molecules A and B, respectively. In the crystal, pairs of N—H···O hydrogen bonds form two independent inversion dimers (Fig. 2) with graph-set notations $R^2_2(14)$ (Bernstein *et al.*, 1995). In addition, each molecule contains an intramolecular O—H···N hydrogen bond with an S(6) notation. Some examples of pyrazoline structures have been published (Fun *et al.*, 2012; Jasinski *et al.*, 2010).

2. Experimental

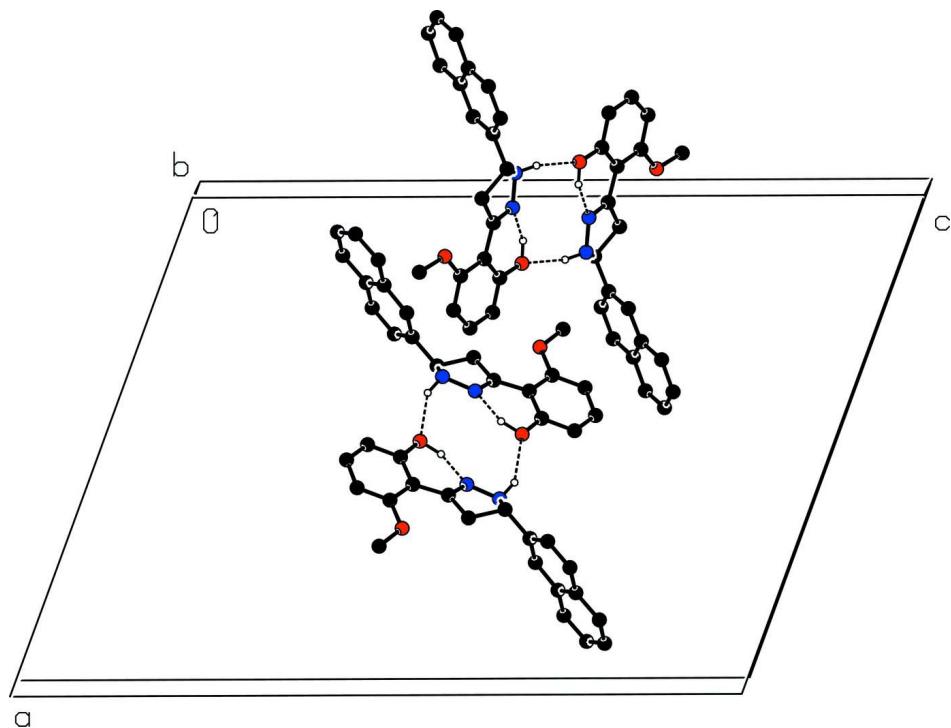
To a solution of 6-methoxy-2-hydroxyacetophenone (10 mmol, 1.66 g) in 50 ml of ethanol was added 2-naphthaldehyde (10 mmol, 1.56 g) and the temperature was adjusted to around 276–277 K in an ice-bath. To the reaction mixture was added 10 ml of 50% (*w/v*) aqueous KOH solution and reaction mixture was stirred at room temperature for 60 h. At the end of the reaction, ice water was added to the mixture and acidified with 6 N HCl (pH = 3–4). The resulting precipitate was filtered and washed with water and ethanol. The crude solid was purified by recrystallization from ethanol to give pure chalcone (m.p.; 403–403 K, yield; 63%). Excess hydrazine monohydrate (1 ml of 64–65% solution, 13 mmol) was added to chalcone compound (5 mmol, 1.52 g) in 30 ml anhydrous ethanol, and the solution was refluxed at 363 K for 3 h. The reaction mixture was cooled to room temperature to yield a solid that was then filtered. The crude solids were purified by recrystallization from ethanol to afford pure pyrazolines (m.p.; 403–403 K, yield; 93%). How were the X-ray quality crystals grown. Repeated recrystallization in ethanol gave colourless needle shape crystals suitable for X-ray diffraction.

3. Refinement

Hydrogen atoms bonded to C atoms were placed in calculated positions with C—H distances ranging from 0.95–1.00 Å and included in the refinement in a riding-model approximation with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ or $1.5U_{\text{eq}}(\text{C}_{\text{methyl}})$. H atoms bonded to N and O atoms were refined independently with isotropic displacement parameters.

**Figure 1**

The asymmetric unit of title compound showing 30% probability ellipsoids.

**Figure 2**

Part of the crystal structure showing hydrogen bonds as dashed lines.

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Crystal data

C₂₀H₁₈N₂O₂
 $M_r = 318.36$
 Monoclinic, P2/c
 Hall symbol: -P 2yc
 $a = 21.0215 (15)$ Å
 $b = 5.6564 (5)$ Å
 $c = 28.785 (2)$ Å
 $\beta = 110.543 (3)^\circ$
 $V = 3205.1 (4)$ Å³
 $Z = 8$

$F(000) = 1344$
 $D_x = 1.320 \text{ Mg m}^{-3}$
 Cu $K\alpha$ radiation, $\lambda = 1.54178$ Å
 Cell parameters from 857 reflections
 $\theta = 3.3\text{--}56.5^\circ$
 $\mu = 0.69 \text{ mm}^{-1}$
 $T = 147 \text{ K}$
 Needle, colourless
 $0.17 \times 0.10 \times 0.07$ mm

Data collection

Bruker Kappa APEX DUO CCD
 diffractometer
 Radiation source: Bruker ImuS
 Multi-layer optics monochromator
 φ and ω scans
 Absorption correction: multi-scan
 (*SADABS*; Bruker, 2012)
 $T_{\min} = 0.695$, $T_{\max} = 0.753$

43387 measured reflections
 5506 independent reflections
 4776 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.045$
 $\theta_{\max} = 66.8^\circ$, $\theta_{\min} = 2.2^\circ$
 $h = -24 \rightarrow 23$
 $k = -6 \rightarrow 6$
 $l = -33 \rightarrow 33$

*Refinement*Refinement on F^2

Least-squares matrix: full

$$R[F^2 > 2\sigma(F^2)] = 0.037$$

$$wR(F^2) = 0.097$$

$$S = 1.05$$

5506 reflections

451 parameters

0 restraints

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of independent and constrained refinement

$$w = 1/[\sigma^2(F_o^2) + (0.0508P)^2 + 0.6182P]$$

where $P = (F_o^2 + 2F_c^2)/3$

$$(\Delta/\sigma)_{\text{max}} = 0.001$$

$$\Delta\rho_{\text{max}} = 0.17 \text{ e \AA}^{-3}$$

$$\Delta\rho_{\text{min}} = -0.20 \text{ e \AA}^{-3}$$

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) etc. and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|------|-------------|------------|-------------|----------------------------------|
| O1A | 0.49628 (5) | 0.5886 (2) | 0.56791 (4) | 0.0409 (3) |
| O2A | 0.34244 (6) | 1.2274 (2) | 0.54722 (4) | 0.0440 (3) |
| N1A | 0.41312 (6) | 0.7105 (2) | 0.48182 (4) | 0.0315 (3) |
| N2A | 0.38431 (6) | 0.7224 (2) | 0.42980 (4) | 0.0326 (3) |
| C1A | 0.39789 (6) | 0.8982 (2) | 0.50121 (5) | 0.0246 (3) |
| C2A | 0.35765 (6) | 1.0709 (2) | 0.46192 (5) | 0.0254 (3) |
| H2AA | 0.3748 | 1.2344 | 0.4698 | 0.030* |
| H2AB | 0.3087 | 1.0666 | 0.4572 | 0.030* |
| C3A | 0.37111 (6) | 0.9740 (2) | 0.41647 (5) | 0.0277 (3) |
| H3AA | 0.4136 | 1.0476 | 0.4149 | 0.033* |
| C4A | 0.31402 (6) | 1.0150 (2) | 0.36773 (5) | 0.0252 (3) |
| C5A | 0.31489 (7) | 1.2249 (2) | 0.34150 (5) | 0.0297 (3) |
| H5AA | 0.3516 | 1.3323 | 0.3544 | 0.036* |
| C6A | 0.26385 (7) | 1.2767 (2) | 0.29782 (5) | 0.0304 (3) |
| H6AA | 0.2657 | 1.4192 | 0.2809 | 0.036* |
| C7A | 0.20822 (7) | 1.1210 (2) | 0.27751 (5) | 0.0267 (3) |
| C8A | 0.15504 (7) | 1.1653 (3) | 0.23166 (5) | 0.0335 (3) |
| H8AA | 0.1560 | 1.3055 | 0.2137 | 0.040* |
| C9A | 0.10281 (7) | 1.0100 (3) | 0.21309 (5) | 0.0379 (4) |
| H9AA | 0.0679 | 1.0421 | 0.1823 | 0.045* |
| C10A | 0.10019 (7) | 0.8025 (3) | 0.23919 (5) | 0.0361 (3) |
| H10A | 0.0632 | 0.6961 | 0.2261 | 0.043* |
| C11A | 0.15082 (7) | 0.7530 (3) | 0.28344 (5) | 0.0305 (3) |
| H11A | 0.1485 | 0.6122 | 0.3008 | 0.037* |
| C12A | 0.20652 (6) | 0.9091 (2) | 0.30362 (5) | 0.0250 (3) |

| | | | | |
|------|--------------|---------------|-------------|------------|
| C13A | 0.26073 (6) | 0.8605 (2) | 0.34863 (5) | 0.0249 (3) |
| H13A | 0.2602 | 0.7180 | 0.3659 | 0.030* |
| C14A | 0.42050 (6) | 0.9222 (2) | 0.55531 (5) | 0.0267 (3) |
| C15A | 0.39484 (7) | 1.0994 (3) | 0.57825 (5) | 0.0312 (3) |
| C16A | 0.42105 (7) | 1.1334 (3) | 0.62929 (5) | 0.0381 (4) |
| H16A | 0.4040 | 1.2561 | 0.6442 | 0.046* |
| C17A | 0.47218 (7) | 0.9862 (3) | 0.65800 (5) | 0.0440 (4) |
| H17A | 0.4907 | 1.0107 | 0.6928 | 0.053* |
| C18A | 0.49680 (7) | 0.8056 (3) | 0.63740 (6) | 0.0437 (4) |
| H18A | 0.5315 | 0.7049 | 0.6579 | 0.052* |
| C19A | 0.47060 (6) | 0.7707 (3) | 0.58629 (5) | 0.0320 (3) |
| C20A | 0.31232 (10) | 1.4005 (3) | 0.56934 (6) | 0.0507 (5) |
| H20A | 0.2743 | 1.4766 | 0.5435 | 0.076* |
| H20B | 0.3463 | 1.5199 | 0.5863 | 0.076* |
| H20C | 0.2957 | 1.3235 | 0.5934 | 0.076* |
| O1B | -0.09997 (5) | -0.95204 (18) | 0.51393 (4) | 0.0354 (2) |
| O2B | -0.06378 (5) | -0.28470 (19) | 0.62119 (4) | 0.0374 (3) |
| N1B | 0.01760 (6) | -0.7754 (2) | 0.55421 (5) | 0.0369 (3) |
| N2B | 0.08805 (6) | -0.7481 (2) | 0.56768 (5) | 0.0386 (3) |
| C1B | -0.00720 (6) | -0.6051 (2) | 0.57246 (5) | 0.0272 (3) |
| C2B | 0.04742 (6) | -0.4300 (2) | 0.59985 (5) | 0.0273 (3) |
| H2BA | 0.0617 | -0.4504 | 0.6362 | 0.033* |
| H2BB | 0.0323 | -0.2649 | 0.5911 | 0.033* |
| C3B | 0.10441 (6) | -0.4987 (3) | 0.58063 (5) | 0.0302 (3) |
| H3BA | 0.0992 | -0.4067 | 0.5498 | 0.036* |
| C4B | 0.17604 (6) | -0.4685 (2) | 0.61734 (5) | 0.0254 (3) |
| C5B | 0.21368 (7) | -0.2659 (2) | 0.61442 (5) | 0.0299 (3) |
| H5BA | 0.1936 | -0.1499 | 0.5897 | 0.036* |
| C6B | 0.27874 (7) | -0.2345 (2) | 0.64672 (5) | 0.0305 (3) |
| H6BA | 0.3032 | -0.0968 | 0.6441 | 0.037* |
| C7B | 0.31015 (6) | -0.4033 (2) | 0.68385 (5) | 0.0257 (3) |
| C8B | 0.37838 (7) | -0.3815 (3) | 0.71720 (5) | 0.0336 (3) |
| H8BA | 0.4044 | -0.2473 | 0.7151 | 0.040* |
| C9B | 0.40695 (7) | -0.5509 (3) | 0.75216 (5) | 0.0365 (4) |
| H9BA | 0.4528 | -0.5350 | 0.7737 | 0.044* |
| C10B | 0.36893 (7) | -0.7484 (3) | 0.75651 (5) | 0.0349 (3) |
| H10B | 0.3887 | -0.8631 | 0.7815 | 0.042* |
| C11B | 0.30344 (7) | -0.7755 (3) | 0.72472 (5) | 0.0294 (3) |
| H11B | 0.2783 | -0.9107 | 0.7277 | 0.035* |
| C12B | 0.27239 (6) | -0.6065 (2) | 0.68752 (5) | 0.0234 (3) |
| C13B | 0.20506 (6) | -0.6342 (2) | 0.65330 (5) | 0.0249 (3) |
| H13B | 0.1797 | -0.7702 | 0.6554 | 0.030* |
| C14B | -0.07952 (6) | -0.6070 (2) | 0.56665 (5) | 0.0260 (3) |
| C15B | -0.10804 (6) | -0.4417 (3) | 0.59036 (5) | 0.0289 (3) |
| C16B | -0.17715 (7) | -0.4442 (3) | 0.58272 (5) | 0.0328 (3) |
| H16B | -0.1959 | -0.3304 | 0.5985 | 0.039* |
| C17B | -0.21851 (7) | -0.6140 (3) | 0.55187 (5) | 0.0362 (3) |
| H17B | -0.2658 | -0.6137 | 0.5464 | 0.043* |
| C18B | -0.19260 (7) | -0.7822 (3) | 0.52909 (5) | 0.0344 (3) |

| | | | | |
|------|--------------|-------------|-------------|------------|
| H18B | -0.2215 | -0.8983 | 0.5084 | 0.041* |
| C19B | -0.12339 (7) | -0.7809 (3) | 0.53664 (5) | 0.0293 (3) |
| C20B | -0.09006 (7) | -0.1221 (3) | 0.64781 (6) | 0.0381 (4) |
| H20D | -0.0534 | -0.0195 | 0.6683 | 0.057* |
| H20E | -0.1093 | -0.2096 | 0.6691 | 0.057* |
| H20F | -0.1256 | -0.0254 | 0.6243 | 0.057* |
| H1OA | 0.4697 (11) | 0.574 (4) | 0.5327 (9) | 0.078 (7)* |
| H1OB | -0.0542 (11) | -0.935 (4) | 0.5237 (8) | 0.069 (6)* |
| H2NA | 0.4147 (8) | 0.653 (3) | 0.4179 (6) | 0.043 (5)* |
| H2NB | 0.1010 (8) | -0.803 (3) | 0.5426 (7) | 0.049 (5)* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|------|-------------|-------------|------------|-------------|------------|-------------|
| O1A | 0.0324 (5) | 0.0480 (7) | 0.0397 (6) | 0.0140 (5) | 0.0092 (5) | 0.0120 (5) |
| O2A | 0.0597 (7) | 0.0477 (7) | 0.0276 (5) | 0.0247 (6) | 0.0192 (5) | 0.0042 (5) |
| N1A | 0.0282 (6) | 0.0323 (7) | 0.0286 (6) | 0.0055 (5) | 0.0032 (5) | -0.0009 (5) |
| N2A | 0.0313 (6) | 0.0335 (7) | 0.0279 (6) | 0.0108 (5) | 0.0041 (5) | -0.0042 (5) |
| C1A | 0.0209 (6) | 0.0250 (7) | 0.0276 (7) | -0.0002 (5) | 0.0083 (5) | 0.0007 (6) |
| C2A | 0.0281 (6) | 0.0246 (7) | 0.0240 (7) | 0.0012 (5) | 0.0097 (5) | 0.0007 (5) |
| C3A | 0.0254 (6) | 0.0298 (7) | 0.0288 (7) | -0.0006 (6) | 0.0106 (5) | -0.0019 (6) |
| C4A | 0.0269 (6) | 0.0286 (7) | 0.0240 (6) | 0.0037 (6) | 0.0137 (5) | -0.0019 (5) |
| C5A | 0.0317 (7) | 0.0281 (7) | 0.0327 (7) | -0.0012 (6) | 0.0157 (6) | -0.0026 (6) |
| C6A | 0.0374 (7) | 0.0254 (7) | 0.0325 (7) | 0.0032 (6) | 0.0176 (6) | 0.0051 (6) |
| C7A | 0.0314 (7) | 0.0277 (7) | 0.0245 (7) | 0.0086 (6) | 0.0142 (6) | 0.0024 (5) |
| C8A | 0.0377 (8) | 0.0341 (8) | 0.0301 (7) | 0.0111 (7) | 0.0137 (6) | 0.0091 (6) |
| C9A | 0.0339 (7) | 0.0470 (9) | 0.0279 (7) | 0.0123 (7) | 0.0047 (6) | 0.0058 (7) |
| C10A | 0.0296 (7) | 0.0398 (9) | 0.0354 (8) | 0.0012 (6) | 0.0067 (6) | -0.0025 (7) |
| C11A | 0.0319 (7) | 0.0287 (7) | 0.0308 (7) | 0.0038 (6) | 0.0109 (6) | 0.0022 (6) |
| C12A | 0.0284 (6) | 0.0259 (7) | 0.0236 (6) | 0.0061 (5) | 0.0129 (5) | 0.0002 (5) |
| C13A | 0.0288 (7) | 0.0256 (7) | 0.0226 (6) | 0.0051 (6) | 0.0121 (5) | 0.0039 (5) |
| C14A | 0.0227 (6) | 0.0321 (7) | 0.0255 (7) | -0.0040 (6) | 0.0085 (5) | 0.0037 (6) |
| C15A | 0.0335 (7) | 0.0348 (8) | 0.0273 (7) | -0.0019 (6) | 0.0134 (6) | 0.0036 (6) |
| C16A | 0.0374 (8) | 0.0508 (10) | 0.0294 (8) | -0.0096 (7) | 0.0159 (6) | -0.0042 (7) |
| C17A | 0.0285 (7) | 0.0790 (13) | 0.0237 (7) | -0.0118 (8) | 0.0083 (6) | 0.0015 (8) |
| C18A | 0.0243 (7) | 0.0737 (12) | 0.0314 (8) | 0.0018 (7) | 0.0076 (6) | 0.0163 (8) |
| C19A | 0.0205 (6) | 0.0432 (9) | 0.0327 (7) | -0.0008 (6) | 0.0097 (6) | 0.0087 (6) |
| C20A | 0.0778 (12) | 0.0473 (10) | 0.0380 (9) | 0.0246 (9) | 0.0341 (9) | 0.0059 (8) |
| O1B | 0.0328 (5) | 0.0364 (6) | 0.0346 (6) | -0.0051 (5) | 0.0088 (4) | -0.0119 (5) |
| O2B | 0.0270 (5) | 0.0447 (6) | 0.0431 (6) | -0.0023 (4) | 0.0156 (4) | -0.0194 (5) |
| N1B | 0.0234 (6) | 0.0434 (8) | 0.0384 (7) | 0.0017 (5) | 0.0038 (5) | -0.0168 (6) |
| N2B | 0.0217 (6) | 0.0481 (8) | 0.0415 (7) | 0.0025 (5) | 0.0054 (5) | -0.0238 (6) |
| C1B | 0.0258 (6) | 0.0315 (8) | 0.0229 (7) | 0.0026 (6) | 0.0067 (5) | -0.0045 (6) |
| C2B | 0.0232 (6) | 0.0295 (7) | 0.0289 (7) | 0.0012 (5) | 0.0085 (5) | -0.0054 (6) |
| C3B | 0.0254 (7) | 0.0395 (8) | 0.0250 (7) | 0.0027 (6) | 0.0079 (6) | -0.0047 (6) |
| C4B | 0.0235 (6) | 0.0317 (7) | 0.0228 (6) | 0.0033 (6) | 0.0105 (5) | -0.0058 (6) |
| C5B | 0.0353 (7) | 0.0260 (7) | 0.0293 (7) | 0.0046 (6) | 0.0123 (6) | -0.0001 (6) |
| C6B | 0.0355 (7) | 0.0238 (7) | 0.0350 (8) | -0.0037 (6) | 0.0160 (6) | -0.0033 (6) |
| C7B | 0.0268 (6) | 0.0262 (7) | 0.0272 (7) | -0.0020 (5) | 0.0135 (5) | -0.0076 (5) |
| C8B | 0.0286 (7) | 0.0359 (8) | 0.0365 (8) | -0.0088 (6) | 0.0116 (6) | -0.0122 (7) |

| | | | | | | |
|------|------------|-------------|------------|-------------|------------|-------------|
| C9B | 0.0257 (7) | 0.0480 (9) | 0.0314 (8) | 0.0018 (7) | 0.0044 (6) | -0.0130 (7) |
| C10B | 0.0352 (7) | 0.0426 (9) | 0.0252 (7) | 0.0086 (7) | 0.0087 (6) | -0.0014 (6) |
| C11B | 0.0324 (7) | 0.0308 (8) | 0.0277 (7) | 0.0012 (6) | 0.0140 (6) | -0.0006 (6) |
| C12B | 0.0244 (6) | 0.0264 (7) | 0.0225 (6) | -0.0006 (5) | 0.0121 (5) | -0.0056 (5) |
| C13B | 0.0241 (6) | 0.0278 (7) | 0.0264 (7) | -0.0042 (5) | 0.0132 (5) | -0.0055 (6) |
| C14B | 0.0242 (6) | 0.0311 (7) | 0.0221 (6) | -0.0002 (6) | 0.0073 (5) | 0.0003 (5) |
| C15B | 0.0266 (7) | 0.0350 (8) | 0.0248 (7) | -0.0008 (6) | 0.0088 (5) | -0.0012 (6) |
| C16B | 0.0267 (7) | 0.0436 (9) | 0.0306 (7) | 0.0030 (6) | 0.0133 (6) | 0.0022 (6) |
| C17B | 0.0251 (7) | 0.0525 (10) | 0.0311 (7) | -0.0027 (7) | 0.0101 (6) | 0.0048 (7) |
| C18B | 0.0294 (7) | 0.0445 (9) | 0.0270 (7) | -0.0084 (6) | 0.0069 (6) | -0.0001 (6) |
| C19B | 0.0317 (7) | 0.0327 (8) | 0.0233 (7) | -0.0017 (6) | 0.0093 (6) | 0.0013 (6) |
| C20B | 0.0360 (8) | 0.0412 (9) | 0.0420 (9) | 0.0031 (7) | 0.0198 (7) | -0.0120 (7) |

Geometric parameters (\AA , $\text{\textcircled{}}^{\circ}$)

| | | | |
|-----------|-------------|-----------|-------------|
| O1A—C19A | 1.3539 (19) | O1B—C19B | 1.3536 (17) |
| O1A—H1OA | 0.97 (2) | O1B—H1OB | 0.91 (2) |
| O2A—C15A | 1.3582 (17) | O2B—C15B | 1.3648 (17) |
| O2A—C20A | 1.4313 (18) | O2B—C20B | 1.4269 (17) |
| N1A—C1A | 1.2910 (18) | N1B—C1B | 1.2915 (18) |
| N1A—N2A | 1.4059 (16) | N1B—N2B | 1.4016 (16) |
| N2A—C3A | 1.4745 (18) | N2B—C3B | 1.469 (2) |
| N2A—H2NA | 0.914 (18) | N2B—H2NB | 0.911 (19) |
| C1A—C14A | 1.4664 (18) | C1B—C14B | 1.4699 (18) |
| C1A—C2A | 1.5091 (18) | C1B—C2B | 1.5121 (18) |
| C2A—C3A | 1.5338 (18) | C2B—C3B | 1.5354 (18) |
| C2A—H2AA | 0.9900 | C2B—H2BA | 0.9900 |
| C2A—H2AB | 0.9900 | C2B—H2BB | 0.9900 |
| C3A—C4A | 1.5104 (18) | C3B—C4B | 1.5155 (18) |
| C3A—H3AA | 1.0000 | C3B—H3BA | 1.0000 |
| C4A—C13A | 1.3742 (19) | C4B—C13B | 1.3706 (19) |
| C4A—C5A | 1.4104 (19) | C4B—C5B | 1.412 (2) |
| C5A—C6A | 1.368 (2) | C5B—C6B | 1.369 (2) |
| C5A—H5AA | 0.9500 | C5B—H5BA | 0.9500 |
| C6A—C7A | 1.416 (2) | C6B—C7B | 1.413 (2) |
| C6A—H6AA | 0.9500 | C6B—H6BA | 0.9500 |
| C7A—C8A | 1.4210 (19) | C7B—C12B | 1.4216 (19) |
| C7A—C12A | 1.4218 (19) | C7B—C8B | 1.4239 (19) |
| C8A—C9A | 1.361 (2) | C8B—C9B | 1.366 (2) |
| C8A—H8AA | 0.9500 | C8B—H8BA | 0.9500 |
| C9A—C10A | 1.405 (2) | C9B—C10B | 1.404 (2) |
| C9A—H9AA | 0.9500 | C9B—H9BA | 0.9500 |
| C10A—C11A | 1.372 (2) | C10B—C11B | 1.368 (2) |
| C10A—H10A | 0.9500 | C10B—H10B | 0.9500 |
| C11A—C12A | 1.4181 (19) | C11B—C12B | 1.4128 (19) |
| C11A—H11A | 0.9500 | C11B—H11B | 0.9500 |
| C12A—C13A | 1.4200 (18) | C12B—C13B | 1.4211 (18) |
| C13A—H13A | 0.9500 | C13B—H13B | 0.9500 |
| C14A—C19A | 1.4070 (19) | C14B—C15B | 1.4096 (19) |
| C14A—C15A | 1.408 (2) | C14B—C19B | 1.4159 (19) |

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|---------------|-------------|---------------|-------------|
| C15A—C16A | 1.390 (2) | C15B—C16B | 1.3909 (18) |
| C16A—C17A | 1.381 (2) | C16B—C17B | 1.387 (2) |
| C16A—H16A | 0.9500 | C16B—H16B | 0.9500 |
| C17A—C18A | 1.371 (3) | C17B—C18B | 1.372 (2) |
| C17A—H17A | 0.9500 | C17B—H17B | 0.9500 |
| C18A—C19A | 1.392 (2) | C18B—C19B | 1.3936 (19) |
| C18A—H18A | 0.9500 | C18B—H18B | 0.9500 |
| C20A—H20A | 0.9800 | C20B—H20D | 0.9800 |
| C20A—H20B | 0.9800 | C20B—H20E | 0.9800 |
| C20A—H20C | 0.9800 | C20B—H20F | 0.9800 |
| | | | |
| C19A—O1A—H1OA | 108.1 (13) | C19B—O1B—H1OB | 107.5 (14) |
| C15A—O2A—C20A | 117.08 (12) | C15B—O2B—C20B | 117.68 (10) |
| C1A—N1A—N2A | 109.97 (11) | C1B—N1B—N2B | 110.08 (11) |
| N1A—N2A—C3A | 107.15 (11) | N1B—N2B—C3B | 107.86 (11) |
| N1A—N2A—H2NA | 106.4 (11) | N1B—N2B—H2NB | 108.9 (11) |
| C3A—N2A—H2NA | 114.3 (11) | C3B—N2B—H2NB | 114.9 (12) |
| N1A—C1A—C14A | 119.87 (12) | N1B—C1B—C14B | 119.81 (12) |
| N1A—C1A—C2A | 111.55 (11) | N1B—C1B—C2B | 111.08 (11) |
| C14A—C1A—C2A | 128.58 (12) | C14B—C1B—C2B | 129.05 (11) |
| C1A—C2A—C3A | 100.66 (10) | C1B—C2B—C3B | 101.06 (10) |
| C1A—C2A—H2AA | 111.6 | C1B—C2B—H2BA | 111.6 |
| C3A—C2A—H2AA | 111.6 | C3B—C2B—H2BA | 111.6 |
| C1A—C2A—H2AB | 111.6 | C1B—C2B—H2BB | 111.6 |
| C3A—C2A—H2AB | 111.6 | C3B—C2B—H2BB | 111.6 |
| H2AA—C2A—H2AB | 109.4 | H2BA—C2B—H2BB | 109.4 |
| N2A—C3A—C4A | 113.94 (11) | N2B—C3B—C4B | 111.84 (11) |
| N2A—C3A—C2A | 101.37 (11) | N2B—C3B—C2B | 101.06 (11) |
| C4A—C3A—C2A | 114.39 (11) | C4B—C3B—C2B | 115.47 (11) |
| N2A—C3A—H3AA | 108.9 | N2B—C3B—H3BA | 109.4 |
| C4A—C3A—H3AA | 108.9 | C4B—C3B—H3BA | 109.4 |
| C2A—C3A—H3AA | 108.9 | C2B—C3B—H3BA | 109.4 |
| C13A—C4A—C5A | 118.88 (12) | C13B—C4B—C5B | 119.33 (12) |
| C13A—C4A—C3A | 122.98 (12) | C13B—C4B—C3B | 121.31 (12) |
| C5A—C4A—C3A | 118.12 (12) | C5B—C4B—C3B | 119.36 (12) |
| C6A—C5A—C4A | 121.26 (13) | C6B—C5B—C4B | 120.80 (13) |
| C6A—C5A—H5AA | 119.4 | C6B—C5B—H5BA | 119.6 |
| C4A—C5A—H5AA | 119.4 | C4B—C5B—H5BA | 119.6 |
| C5A—C6A—C7A | 120.92 (13) | C5B—C6B—C7B | 121.10 (13) |
| C5A—C6A—H6AA | 119.5 | C5B—C6B—H6BA | 119.5 |
| C7A—C6A—H6AA | 119.5 | C7B—C6B—H6BA | 119.5 |
| C6A—C7A—C8A | 122.70 (13) | C6B—C7B—C12B | 118.54 (12) |
| C6A—C7A—C12A | 118.45 (12) | C6B—C7B—C8B | 122.99 (13) |
| C8A—C7A—C12A | 118.83 (13) | C12B—C7B—C8B | 118.46 (13) |
| C9A—C8A—C7A | 120.96 (13) | C9B—C8B—C7B | 120.95 (14) |
| C9A—C8A—H8AA | 119.5 | C9B—C8B—H8BA | 119.5 |
| C7A—C8A—H8AA | 119.5 | C7B—C8B—H8BA | 119.5 |
| C8A—C9A—C10A | 120.46 (13) | C8B—C9B—C10B | 120.43 (13) |
| C8A—C9A—H9AA | 119.8 | C8B—C9B—H9BA | 119.8 |

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|----------------|-------------|----------------|-------------|
| C10A—C9A—H9AA | 119.8 | C10B—C9B—H9BA | 119.8 |
| C11A—C10A—C9A | 120.27 (14) | C11B—C10B—C9B | 120.01 (14) |
| C11A—C10A—H10A | 119.9 | C11B—C10B—H10B | 120.0 |
| C9A—C10A—H10A | 119.9 | C9B—C10B—H10B | 120.0 |
| C10A—C11A—C12A | 120.87 (13) | C10B—C11B—C12B | 121.32 (13) |
| C10A—C11A—H11A | 119.6 | C10B—C11B—H11B | 119.3 |
| C12A—C11A—H11A | 119.6 | C12B—C11B—H11B | 119.3 |
| C11A—C12A—C13A | 122.43 (12) | C11B—C12B—C13B | 122.25 (12) |
| C11A—C12A—C7A | 118.60 (12) | C11B—C12B—C7B | 118.81 (12) |
| C13A—C12A—C7A | 118.97 (12) | C13B—C12B—C7B | 118.94 (12) |
| C4A—C13A—C12A | 121.50 (12) | C4B—C13B—C12B | 121.28 (12) |
| C4A—C13A—H13A | 119.2 | C4B—C13B—H13B | 119.4 |
| C12A—C13A—H13A | 119.2 | C12B—C13B—H13B | 119.4 |
| C19A—C14A—C15A | 117.46 (13) | C15B—C14B—C19B | 117.35 (12) |
| C19A—C14A—C1A | 120.57 (12) | C15B—C14B—C1B | 122.59 (12) |
| C15A—C14A—C1A | 121.95 (12) | C19B—C14B—C1B | 120.05 (12) |
| O2A—C15A—C16A | 123.43 (13) | O2B—C15B—C16B | 123.04 (12) |
| O2A—C15A—C14A | 115.27 (12) | O2B—C15B—C14B | 115.96 (11) |
| C16A—C15A—C14A | 121.28 (14) | C16B—C15B—C14B | 120.99 (13) |
| C17A—C16A—C15A | 119.08 (15) | C17B—C16B—C15B | 119.57 (13) |
| C17A—C16A—H16A | 120.5 | C17B—C16B—H16B | 120.2 |
| C15A—C16A—H16A | 120.5 | C15B—C16B—H16B | 120.2 |
| C18A—C17A—C16A | 121.53 (14) | C18B—C17B—C16B | 121.40 (13) |
| C18A—C17A—H17A | 119.2 | C18B—C17B—H17B | 119.3 |
| C16A—C17A—H17A | 119.2 | C16B—C17B—H17B | 119.3 |
| C17A—C18A—C19A | 119.57 (14) | C17B—C18B—C19B | 119.32 (14) |
| C17A—C18A—H18A | 120.2 | C17B—C18B—H18B | 120.3 |
| C19A—C18A—H18A | 120.2 | C19B—C18B—H18B | 120.3 |
| O1A—C19A—C18A | 117.18 (13) | O1B—C19B—C18B | 117.24 (12) |
| O1A—C19A—C14A | 121.87 (13) | O1B—C19B—C14B | 121.46 (12) |
| C18A—C19A—C14A | 120.94 (14) | C18B—C19B—C14B | 121.30 (13) |
| O2A—C20A—H20A | 109.5 | O2B—C20B—H20D | 109.5 |
| O2A—C20A—H20B | 109.5 | O2B—C20B—H20E | 109.5 |
| H20A—C20A—H20B | 109.5 | H20D—C20B—H20E | 109.5 |
| O2A—C20A—H20C | 109.5 | O2B—C20B—H20F | 109.5 |
| H20A—C20A—H20C | 109.5 | H20D—C20B—H20F | 109.5 |
| H20B—C20A—H20C | 109.5 | H20E—C20B—H20F | 109.5 |

Hydrogen-bond geometry (Å, °)

| D—H···A | D—H | H···A | D···A | D—H···A |
|------------------------------|------------|------------|-------------|------------|
| O1A—H1OA···N1A | 0.97 (2) | 1.71 (2) | 2.5754 (15) | 145 (2) |
| O1B—H1OB···N1B | 0.91 (2) | 1.71 (2) | 2.5377 (15) | 149 (2) |
| N2A—H2NA···O1A ⁱ | 0.914 (18) | 2.234 (18) | 3.0470 (16) | 147.7 (15) |
| N2B—H2NB···O1B ⁱⁱ | 0.911 (19) | 2.131 (19) | 2.9787 (16) | 154.4 (15) |

Symmetry codes: (i) -x+1, -y+1, -z+1; (ii) -x, -y-2, -z+1.